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INCREASING THE LASER INDUCED DAMAGE THRESHOLD OF SINGLE CRYSTAL $ZnGeP_2$ (PREPRINT)

Kevin T. Zawilski, Scott D. Setzler, Peter G. Schunemann, and Thomas M. Pollak

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14. ABSTRACT
The laser induced damage threshold (LIDT) of single crystal zinc germanium phosphide (ZGP), $ZnGeP_2$, was increased to 2 J/cm^2 at 2.05 μm and 10 kHz pulse rate frequency (double the previously measured value of 1 J/cm^2). This increased LIDT was achieved by improving the polishing of ZGP OPO crystals. The surface preparation of ZGP samples was determined to be of great importance because laser-induced damage has been observed to always initiate at the surface rather than in the bulk of the material. Two different polishing techniques were evaluated by comparing the surfaces of ZGP samples fabricated in the same manner apart from the polishing stage. Surfaces were characterized using scanning white light interferometry (SWLI) in order to determine RMS surface roughness and sample flatness. The photon backscatter technique (PBS) was used to determine the degree of surface and subsurface damage in the sample induced through the fabrication process. Both uncoated and anti-reflection coated samples were damage tested. The effect of subsurface damage in the samples was studied by removing different amounts of material during polishing for otherwise identical samples. The amount of material removed was correlated to the observed LIDT. Statistical LIDT was measured using a high-average-power, repetitively Q-switched Tm,Ho:YLF 2.05-μm pump laser. On average, lower surface roughness and photon backscatter measurements was a good indicator of ZGP samples exhibiting higher LIDT. The removal of more material during polishing significantly improved the LIDT of otherwise identical samples, indicating the importance of subsurface damage defects in the LIDT of ZGP.

15. SUBJECT TERMS
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ABSTRACT

The laser induced damage threshold (LIDT) of single crystal zinc germanium phosphide (ZGP), $ZnGeP_2$, was increased to 2 J/cm^2 at 2.05 μm and 10 kHz pulse rate frequency (double the previously measured value of 1 J/cm^2). This increased LIDT was achieved by improving the polishing of ZGP OPO crystals. The surface preparation of ZGP samples was determined to be of great importance because laser-induced damage has been observed to always initiate at the surface rather than in the bulk of the material. Two different polishing techniques were evaluated by comparing the surfaces of ZGP samples fabricated in the same manner apart from the polishing stage. Surfaces were characterized using scanning white light interferometry (SWLI) in order to determine RMS surface roughness and sample flatness. The photon backscatter technique (PBS) was used to determine the degree of surface and subsurface damage in the sample induced through the fabrication process. Both uncoated and anti-reflection coated samples were damage tested. The effect of subsurface damage in the samples was studied by removing different amounts of material during polishing for otherwise identical samples. The amount of material removed was correlated to the observed LIDT. Statistical LIDT was measured using a high-average-power, repetitively Q-switched Tm,Ho:YLF 2.05-μm pump laser. On average, lower surface roughness and photon backscatter measurements was a good indicator of ZGP samples exhibiting higher LIDT. The removal of more material during polishing significantly improved the LIDT of otherwise identical samples, indicating the importance of subsurface damage defects in the LIDT of ZGP.

Keywords: laser induced damage threshold, mid-infrared materials, non-linear optic materials, zinc germanium phosphide, surface characterization, single crystal

1. INTRODUCTION

Zinc germanium phosphide (ZGP), $ZnGeP_2$, is a non-linear optical crystal with excellent properties for laser frequency conversion in the 2-8 μm spectral range including its high non-linear coefficient (d_{14}=75 pm/V) and thermal conductivity (0.35 W/cm K).[1-5] The potential of ZGP for use in infrared optics has been known for many years,[3-5] however, it has only been relatively recently that large, crack-free single crystals of device quality material have been produced.[2] Because ZGP is now finding its way into an increasing number of applications with more and more demanding requirements, the laser induced damage threshold (LIDT) of ZGP has been of increasing importance.

A study by Peterson et al.[6] during the early development of ZGP remains the only widely-available systematic study on the LIDT of this material to date. Because the study occurred early in the development of ZGP, the size and number of available samples was limited, resulting in a limited statistical sample size. In the study, the least fluence failure method was used where each test site was irradiated with increasingly higher fluences until damage occurred, and the highest fluence level prior to the observation of damage was labeled the damage threshold. The study was performed with a 2.08 μm laser at 1 Hz repetition rates. The resulting damage thresholds ranged from 2-20 J/cm^2 on anti-reflection (AR) coated ZGP samples and varied widely both between sites on a given sample and between samples. The study determined that damage in ZGP was governed by the energy density (J/cm^2) rather than the power density (MW/cm^2) and that damage always occurred at the sample surface rather than in the bulk.

Setzler et al.[7] had performed LIDT tests using the S-on-1 method, which uses a 0% probability for damage as the definition of laser damage threshold. The LIDT using this more rigorous definition was significantly lower than the results of Peterson et al.[6] The LIDT for AR-coated single crystal ZGP optical parametric oscillator (OPO) samples was found to be ~2 J/cm^2 for a 2.09 μm wavelength and 2 Hz repetition rates and ~1 J/cm^2 for a 2.05 μm wavelength and 10 kHz repetition rates. The higher frequency (10 kHz) repetition rates are much more typical of what the OPO would encounter during standard operation. No temperature dependence for LIDT was

observed for the 10 kHz samples over the range of -5ºC to 55ºC. The LIDT was found to increase as the wavelength increased at 10 kHz operation (~1.9 J/cm^2 for 3 μm wavelength and ~2.6 J/cm^2 for 4 μm wavelength). In both studies,[6,7] the LIDT was found to be significantly lower for uncoated ZGP than AR-coated samples, and the LIDT was found to be independent of the bulk ZGP absorption at 2 μm.

Because laser-induced damage was observed to always initiate at the surface rather than in the bulk of the material,[6,7] the surface preparation of ZGP samples was determined to be of great importance. This study quantitatively examines the effect of surface preparation on the LIDT of ZGP in order to determine how the LIDT at 2 μm can be improved. The process of fabricating laser quality ZGP samples from a grown crystal involves several steps that can induce damage in the surface, including cutting the samples from the boule, grinding the samples to shape, and polishing the optical surfaces to laser quality. The dependence of LIDT on two aspects of sample fabrication was examined in this study. First, two different polishing techniques were evaluated. The surfaces of ZGP samples fabricated in the same manner apart from the polishing stage were quantitatively examined using scanning white light interferometry (SWLI) and the photon backscatter (PBS) technique. The LIDT was then measured for each part using the S-on-1 method in order to determine any correlation between the surface measurements and the LIDT. Second, the effect of subsurface damage on LIDT was evaluated by removing different amounts of material during polishing for otherwise identical samples. The amount of material removed was correlated to the observed LIDT.

2. EXPERIMENTAL PROCEDURE

2.1 Sample Preparation

ZGP OPO samples were fabricated from single crystal boules grown by the horizontal gradient freeze (HGF) method in transparent furnaces under low temperature gradients as described by Schunemann et al.[2,8] A picture of a typical ZGP single crystal boule along with typical OPO samples is shown in Fig. 1. Samples were cut from several different boules and measured 6 mm x 6 mm x 13 mm. The 6 mm x 6 mm faces were polished to a laser optic quality finish using either an

older polishing technique (polish 1) or a newly developed technique (polish 2). Twelve samples were fabricated identically except for the polish. Half of the samples used polish 1 and the other half used polish 2. Two samples from each polish type were left uncoated, and the remaining samples were AR-coated prior to damage testing.

The polished surfaces were characterized using SWLI and PBS prior to the application of any AR-coating. SWLI was used to measure RMS surface roughness and the peak to valley surface flatness. RMS surface roughness has been linked by varying degrees to LIDT.[9-13] House et al.[9] found that RMS surface roughness was a good indicator of the LIDT of fused silica, and more recently Randi et al.[10] have found that RMS surface roughness was a good indicator of the depth of subsurface damage caused by polishing in some single crystalline optical materials, including: silicon, lithium niobate, calcium fluoride, magnesium fluoride, and sapphire. Camp et al.[11] found that subsurface damage affected the LIDT of fused silica, but also found that RMS roughness was only weakly linked to subsurface damage and was not a good indicator of LIDT. The surface flatness measurements were used as a general indicator of surface quality. PBS measurements were performed by VTI, Inc.[14] Along with RMS roughness; PBS measurements were used as a non-destructive measurement of surface and subsurface damage. The PBS technique involves a 632.8 nm probe laser with a spot size of 0.25 mm and 20 mW powers impinging on the sample surface. Photon backscatter is measured using a detector near the path of the incident beam. Magee et al.[15] have shown that PBS measurements are linked to both subsurface defects and laser damage in fused silica. The penetration depth of the PBS laser was estimated to be 0.05 μm in ZGP,[14] which is within the uppermost 'polished' defect layer which can extend from 0.1 to 1 μm according to Camp et al.[11] Subsurface defects from polishing/grinding are estimated to extend to depths up to 100 μm and deformed or strained layers are estimated to extend to depths of 200 μm.[11] The relatively shallow penetration depth made the PBS measurements much more of an indicator of surface scatter rather than subsurface defects in ZGP.

Subsurface damage includes defects that are located below the visible surface and were produced during sample fabrication. In practice, subsurface damage is eliminated or reduced by using progressively gentler polishing steps, making sure that each step removes damage caused by

the previous step. In order to better determine the extent of subsurface damage in the ZGP samples as well as its impact on the LIDT, four samples were prepared using different initial fabrication steps and identical polishes. The initial OPO fabrication steps remove the most material and use the largest grit sizes, and therefore are most likely to produce the deepest subsurface damage in the samples. The first fabrication step in two of the samples was a single pass saw cut, and the other two used a multiple pass machine grind which only removed a small amount of material with each pass. Both the saw blade and the grinding wheel were imbedded with the same size diamond grit. The second fabrication step for two of the samples (one from each of the first step groups) removed 200 μm of material with a 5μm grit, and 500 μm of material was removed with a 5μm grit for the other two samples. The remaining polishing steps for the four samples were identical.

2.2 Laser Damage Testing

Laser damage testing was performed using the S-on-1 testing method (ISO 11254). A high-average-power, repetitively Q-switched Tm,Ho:YLF 2.05-μm laser operating with 15 ns pulse widths at 10 kHz pulse rate frequency and 4 W maximum average power was used as the damage inducing source. The $1/e^2$ beam diameter was focused to 130 μm at the sample surface, and M^2 = 1.1. The beam was periodically recharacterized during the period of damage testing in order to ensure constant beam quality. The test samples were placed into a 4-axis translation stage as shown in Fig. 2. The x, y, and z axes allowed for translation between test sites on a given sample and for the face of the test sample to always be located at the plane of the focused beam waist. The 4^{th} axis allowed for translation between the beam and microscope examination stage.

A close-packed array of test sites was generated to cover the 6 mm x 6 mm surface as shown in Fig. 3a. Spacing between the test sites was 460 μm. Test sites were exposed to the laser for 30 s intervals and were examined with the microscope both prior to and after each exposure. The initial test site near the upper right corner (labeled ref in Fig. 3a) was tested by the least fluence failure method in order to estimate the initial fluence level for testing. The reference site was exposed to increasing fluence levels until damage was observed, and this damaging fluence was chosen as the initial fluence level for S-on-1 testing. The remaining test sites were organized into 10 groups

having 10 test sites each (8 sites in the 10^{th} group due to lack of space). The sites in each group were randomly distributed across the test surface as shown in Fig. 3a. Each site was exposed only once with sites in each group being exposed to the same fluence level. The fraction of damaged sites at each fluence was plotted versus fluence, and the LIDT was determined by the x-intercept of a linear fit to the data. At least two fluence levels with no sites damaged were found for each sample in order to ensure that low probability damage mechanisms were not missed. A photograph of a typical sample surface after damage testing is shown in Fig 3b.

3. RESULTS AND DISCUSSION

3.1. Surface characterization of ZGP samples

The average results of the surface characterization measurements are given in Table 1. Both the RMS surface roughness and sample flatness values given in Table 1 are the measurements averaged over the six parts from each polish type, and both of these measurements are significantly better for the newly developed polish. Polish 2 also has a much smaller deviation over the six samples, making the polish more consistent. The average PBS measurement for a given surface varied widely between samples. The low end of the range for both polish types was near 30 PPM/Sr, and several surfaces from each polish type had average PBS values near this value. Several samples prepared by both polishes also exhibited significantly higher scatter. Polish 1 had samples covering a much larger range of PBS scatter (30 – 9500 PPM/Sr) compared to polish 2 (30 – 250 PPM/Sr), demonstrating again that polish 2 gave a more consistent finish. Overall, the newly developed polish created flatter, smoother surfaces that scattered less light than the older polish.

3.2. Laser induced damage threshold of ZGP samples

Besides the visual indicator of damage as observed through the microscope, laser damage was often accompanied by other visual and aural indicators, most commonly, a flash of light and a high pitched 'pinging' sound. The onset of damage could also be monitored via the transmitted power through the crystal, as any damage always resulted in a drop in transmitted power. Laser damage in the AR-coated samples most often occurred very quickly (within the first second or first 10,000 pulses). However, in a few cases, damage took anywhere between a few seconds to sometimes more than 20 s (after more than 200,000 pulses) to become apparent. In almost every case regarding the AR-coated samples, damage occurred suddenly with a dramatic decrease in transmitted power. In the uncoated samples, evidence of damaging could sometimes be observed by a gradual decrease in transmitted power before any visual damage could be seen optically with the microscope. This was typically observed when the fluence was near the LIDT of the sample. Transmitted power would continue to decrease over several seconds until a flash was seen and a 'pinging' sound could be heard, at which point visible damage would be evident when the surface was observed with the microscope.

Microscope images of some typical laser damage sites are shown in Fig. 4. In AR-coated samples, damage was most often seen at the entrance face, and damage was rarely observed at the exit face. However, in the uncoated samples, damage could be observed regularly on both faces near the LIDT. As fluence increased past the damage threshold, damage was more likely to occur on the entrance face. In a couple of instances, very close to the LIDT of the sample, minor damage could be seen on the entrance face as shown in Fig. 4a. However, entrance face damage more typically appeared in the form of a crater on the surface with ejecta surrounding it as seen in Fig. 4b and Fig. 4c. As the fluence was increased past the LIDT, the relative size of the crater would increase. Exit face damage was observed to be a bulge on the exit face, and appeared to be more of a distortion of the surface as shown in Fig. 4d, rather than the craters observed on the entrance face.

3.2.1 Comparison of polishing techniques

Fig. 5 shows a damage frequency plot with typical results from both polishes both with and without AR-coatings. Dashed lines are fits to the uncoated sample data, and solid lines are fits to the AR-coated sample data. Polish 2 typically had a higher LIDT than polish 1 for both coated and

uncoated samples. AR-coated samples had higher damage thresholds than uncoated samples, and the slope of the damage frequency plot was much steeper for AR-coated parts. The increased slope indicates a more even distribution of surface defects compared to uncoated samples. This suggests that the AR-coating or something during the AR-coating process creates a more uniform distribution of damaging defects.

Table 2 gives the average LIDT results for each polish both with and without AR-coatings. The new polish has a higher average LIDT in both coated and uncoated samples. Uncoated samples have a consistently lower LIDT than their AR-coated counterparts. Both polishes show an improved LIDT over that reported by Setzler et al.[7] (1.0 J/cm^2) with the same AR-coating and under the same operating conditions of 2.05 μm and 10 kHz.

Fig. 6 shows a plot comparing the non-destructive surface characterization measurements to the measured LIDT for each sample. Several additional samples prepared by an alternate polishing technique (not previously discussed, but characterized and damage tested in the same manner as described previously) are included in this figure in order to have a more complete data set. In both Fig. 6a and Fig. 6b, diamonds represent AR-coated samples, and squares represent uncoated samples. A trendline is drawn only for the AR-coated samples. Fig. 6a shows a plot comparing RMS surface roughness to LIDT for each sample. A trend is present showing that as RMS roughness increases, LIDT decreases. Fig. 6b shows a similar plot comparing PBS measurements (on a log scale) to LIDT for each sample. A similar trend is present showing a correlation between decreased average PBS magnitude and increased LIDT. A large amount of scatter is present in the data for both Fig. 6a and Fig 6b, making the RMS roughness and PBS measurements not a good indicator of LIDT on a part to part basis. However, when the RMS roughness and PBS measurements are averaged over several parts for a given polishing technique (Table 1), and are then compared with average LIDT for the same surface preparations (Table 2), these non-destructive characterization techniques appear to be a good indicator of improvements to LIDT.

3.2.2 Comparison of subsurface damage effects

Because polish 2 was found to be the superior polish, the four subsurface damage testing samples were prepared using this polish. In order to examine only the effects surface preparation in the absence of any possible effects created by the AR-coating, the entrance faces of these samples were left uncoated. In order to eliminate possible differences between entrance and exit face damage, the exit faces of all four samples were prepared identically and were AR-coated.

Fig. 7 shows the damage frequency plots obtained from the four samples with different initial fabrication steps. Fig. 7a and 7c show data from samples that had a single pass saw cut as their first processing step, while Fig. 7b and 7d show data from samples that had a multiple pass machine grind as their first processing step. Fig. 7a and 7b show data from samples that had 200μm of material removed in their second processing step, and Fig. 7c and 7d show data from samples that had 500μm of material removed in their second processing step. The data shows that samples having a first step consisting of the machine grind and identical second steps had a higher damage threshold than their saw cut counterparts, indicating that the machine grind induces less subsurface damage. Removing 500μm rather than 200μm of material in the second step both increases the LIDT by a significant amount and increases the slope of the damage frequency curve. The removal of 200μm of material in the second step clearly does not eliminate all of the subsurface damage produced by either of the initial fabrication steps. The steeper slope produced by removing 500μm in the second step indicates a more uniform distribution of damaging defects.

This data suggests that subsurface damage does play a significant role in the LIDT of ZGP samples. By using a multiple pass machine grind for the first step along with removing 500 μm of material in the second step, a LIDT of >2J/cm^2 was observed for an uncoated ZGP surface. Most likely, the LIDT of ZGP could be increased further by focusing on removing subsurface damage in the samples.

4. CONCLUSIONS

The laser induced damage threshold for ZGP samples with an AR-coating was improved to 2 J/cm^2 from a previously reported value[7] of 1 J/cm^2 under laser operating conditions of 2.05 μm and 10 kHz. The increased threshold was accomplished by improving the surface polish of ZGP OPO samples.

Average RMS surface roughness and PBS measurements proved to be a good non-destructive indicator of general surface quality. The scatter in the RMS surface roughness data and PBS measurements when correlated to the LIDT is quite large on a sample to sample basis. However, when the RMS roughness and PBS measurements are averaged over several samples for a given polish type, and are then compared to the average LIDT for the same surface preparation, these two non-destructive characterization techniques are a good method of distinguishing relative LIDT of different polishes.

The removal of more material during polishing significantly improved the LIDT of otherwise identical samples, indicating the importance of subsurface damage defects in the LIDT of ZGP. The most promising avenue for further increasing the LIDT of ZGP samples is by studying methods to further reduce or eliminate subsurface damage in the samples.

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Tables

Table 1. Average Surface Characterization Results for ZGP Parts

Measurement	***Polish 1 (Old)***	***Polish 2 (New)***
RMS Roughness (nm)	1.2 +/- 0.5	0.57 +/- 0.07
Surface Flatness – Peak to Valley (nm)	225 +/- 90	41 +/- 17
PBS Average Value for a Single Surface (PPM/Sr)	Large Range Low end - 30 High end - 9500	Smaller Range Low end - 30 High end - 250

Table 2. Average Laser Damage Threshold Results for ZGP Parts at 2.05 μm

Coating Type	***Polish 1 (Old)***	***Polish 2 (New)***
Uncoated	0.77 J/cm^2	1.4 J/cm^2
AR-coated	1.4 J/cm^2	2.0 J/cm^2

Figure Captions

Figure 1. Photograph of ZGP single crystalline boule and OPO samples.

Figure 2. Schematic of the laser damage test setup.

Figure 3. a) Schematic of test site matrix distributed on a 6 mm x 6 mm ZGP test surface. Each number represents a series of sites tested at the same fluence level. b) Photograph of a typical 6 mm x 6 mm ZGP test surface post damage testing.

Figure 4. Photographs showing the morphology of damage sites. a), b), and c) show morphologies of entrance face damage sites. At fluences neat the LIDT, minor entrance face damage could sometimes be observed as shown in a). Craters as shown in b) and c) were much more typical entrance face damage morphologies, with craters increasing in size as the fluence was increased. Typical exit face damage, consisting of a bulge rather than a crater, is shown in d).

Figure 5. Damage frequency plot of typical results from both AR-coated and uncoated ZGP samples prepared by both polish 1 and polish 2.

Figure 6. a) Plot comparing the LIDT to the RMS surface roughness for ZGP samples. b) Plot comparing the LIDT to the PBS measurements for ZGP samples.

Figure 7. Plots showing the effect of changing parameters in the initial fabrication steps on the LIDT of ZGP. Parameters were changed in the preparation of the entrance faces of the samples which were left uncoated. Each of the samples had identically prepared, AR-coated exit faces. In plots a) and c) the first fabrication step was a single pass saw cut on the face, and in plots b) and d) the first fabrication step was a multiple pass machine grind with only a small amount of material removed per pass. The second fabrication step involved removing 200 μm of material with a 5 μm grit in plots a) and b), and the second fabrication step involved removing 500 μm of material with a 5 μm grit in plots c) and d).


MILLIMETERS
BAE SYSTEMS
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150
ZnGeP2

Fig. 1

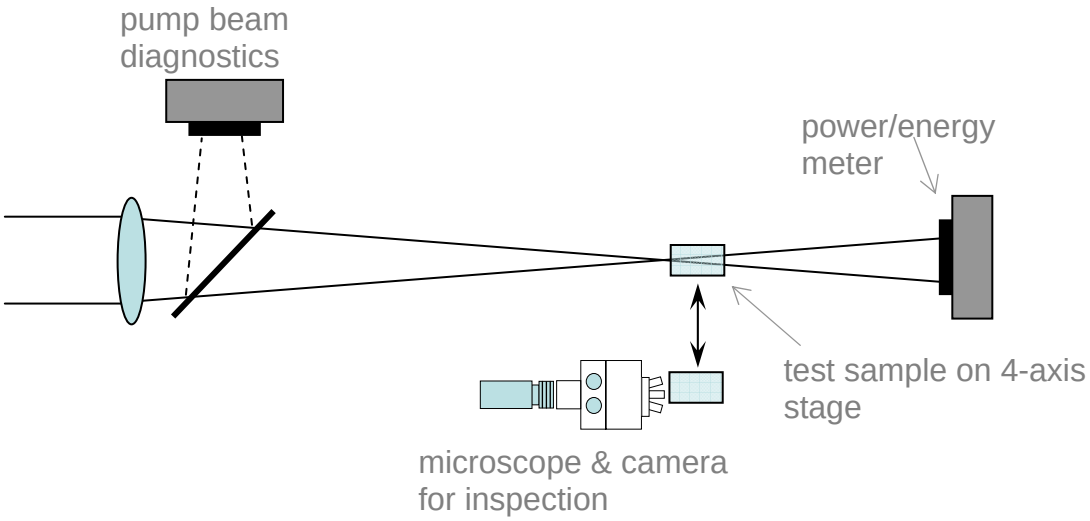
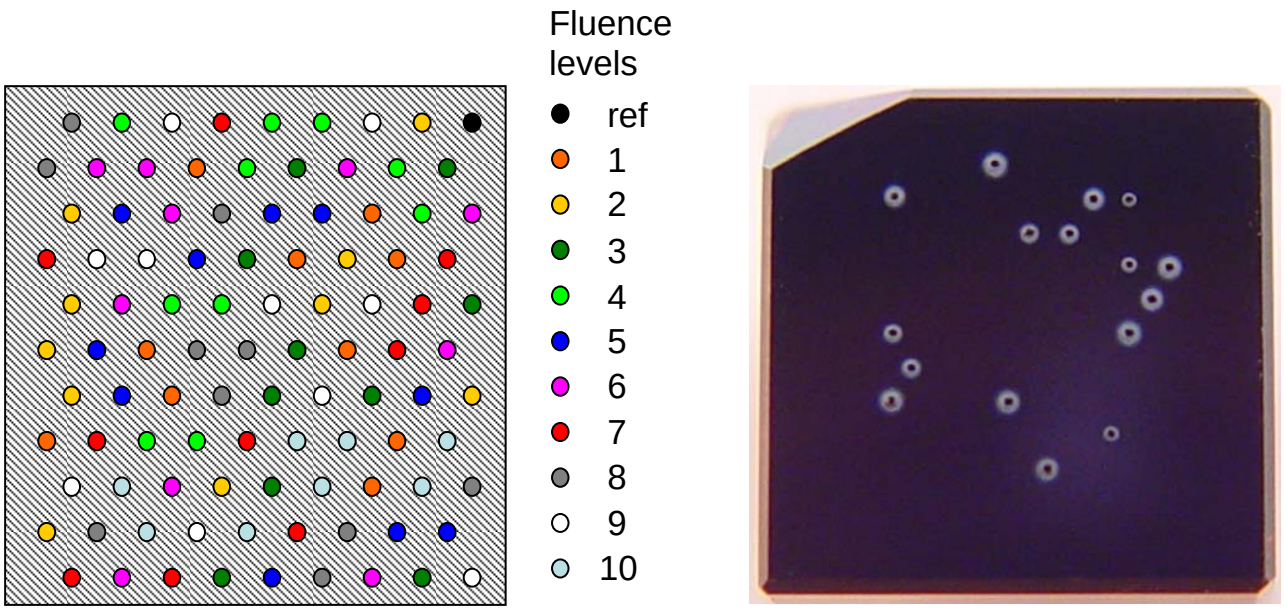

pump beam
diagnostics
power/energy
meter
test sample on 4-axis
stage
microscope & camera
for inspection

Fig. 2



a) b)

Fig. 3

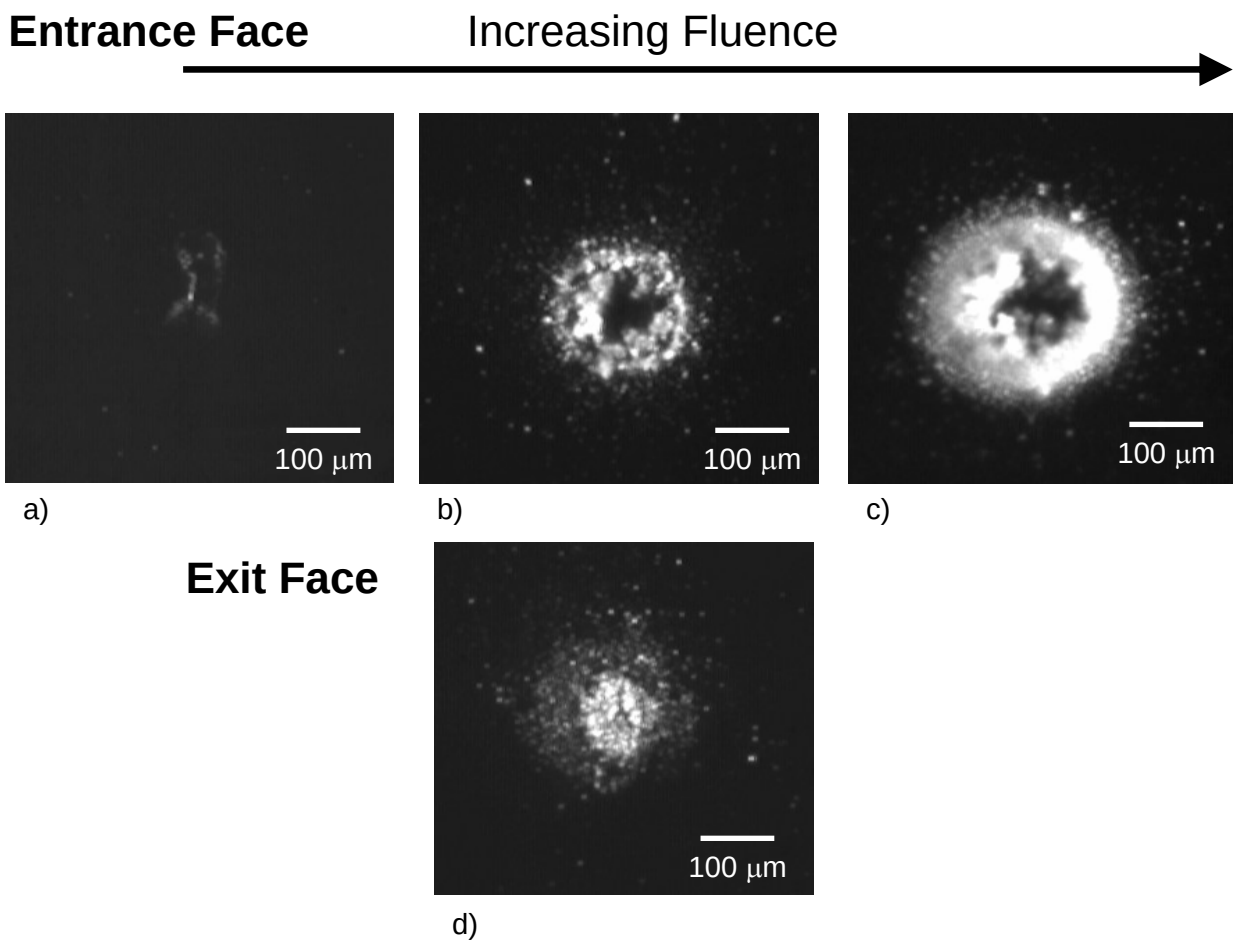

Entrance Face
Increasing Fluence
100 μm
100 μm
100 μm
a)
b)
c)
Exit Face
100 μm
d)

Fig. 4

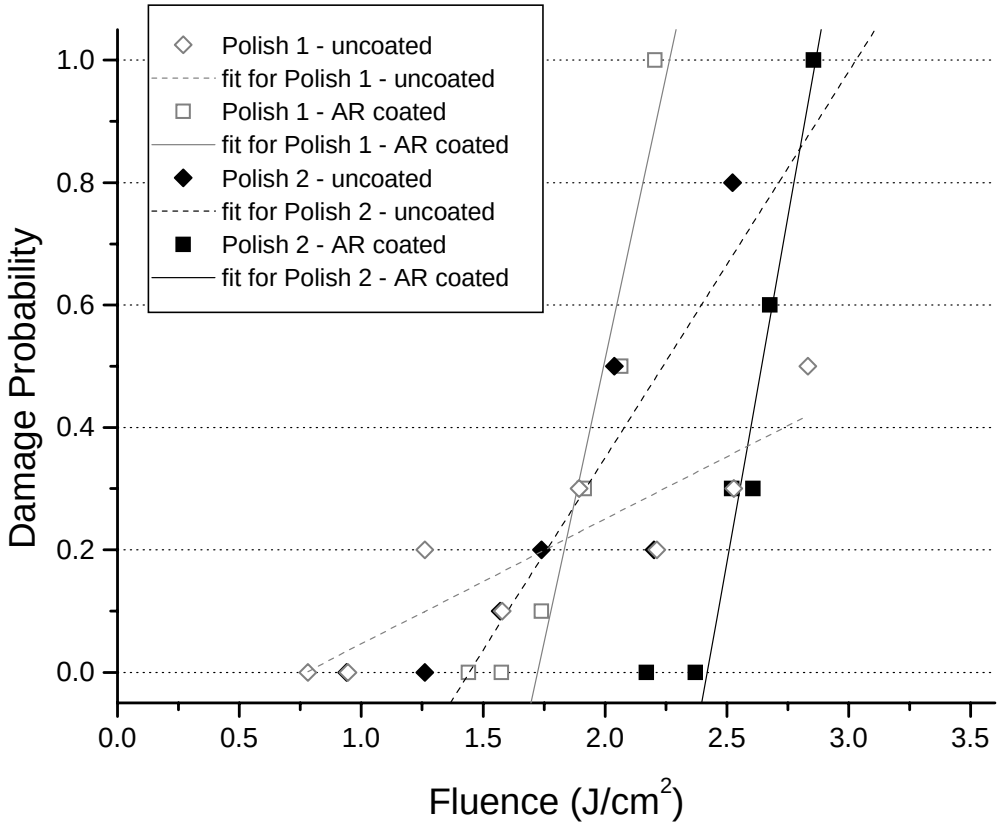
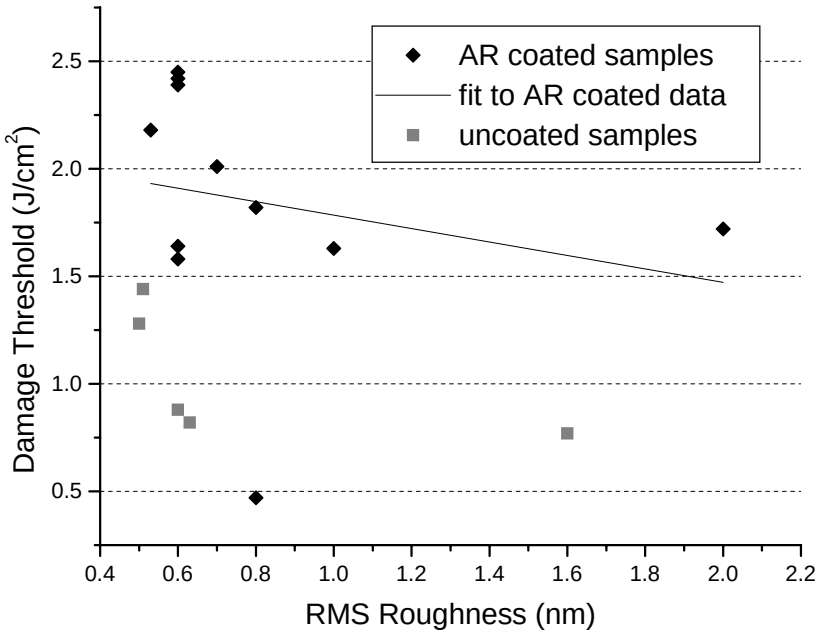

Polish 1 - uncoated
fit for Polish 1 - uncoated
Polish 1 - AR coated
fit for Polish 1 - AR coated
Polish 2 - uncoated
fit for Polish 2 - uncoated
Polish 2 - AR coated
fit for Polish 2 - AR coated
Damage Probability
1.0
0.8
0.6
0.4
0.2
0.0
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
Fluence (J/cm2)

Fig. 5

a)


AR coated samples
fit to AR coated data
uncoated samples
Damage Threshold (J/cm^2)
RMS Roughness (nm)
2.5
2.0
1.5
1.0
0.5
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2

b)

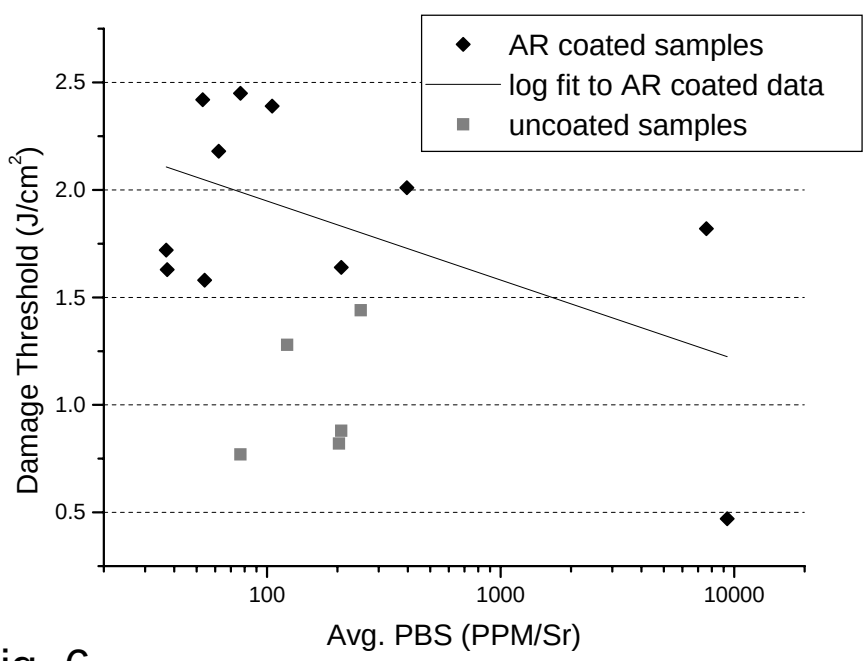

AR coated samples
log fit to AR coated data
uncoated samples
Damage Threshold (J/cm^2)
Avg. PBS (PPM/Sr)
2.5
2.0
1.5
1.0
0.5
100
1000
10000

Fig. 6

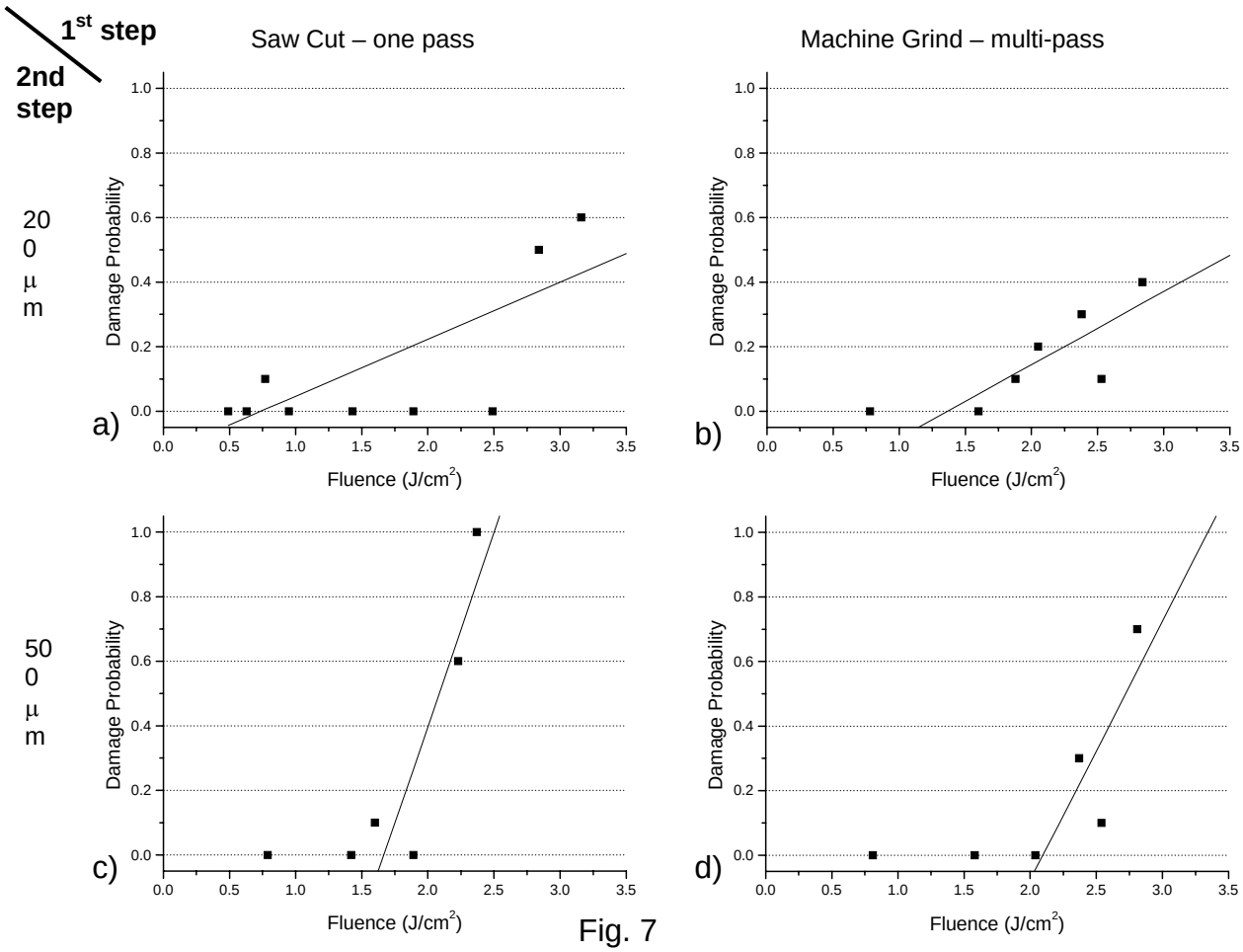

1st step
2nd step
Saw Cut – one pass
Machine Grind – multi-pass
200 µm
500 µm
Damage Probability
Fluence (J/cm2)
a)
b)
c)
d)

Fig. 7